Combustion Analysis for Carbon, Hydrogen, Nitrogen (CHN) and Sulfur (S) or Oxygen (O)

This method is used for the quantitative determination of carbon, hydrogen, and nitrogen (CHN) and sulfur (S) or oxygen (O) in solids or nonvolatile liquids.

Principle of Technique

For CHN analyses, samples are enclosed in tin or copper capsules and dropped into a furnace at 900–950°C. Helium is used as the carrier gas, and oxygen is introduced into the combustion zone. The resulting combustion products are detected either in infrared cells or in thermal conductivity detectors. Carbon is determined as CO_2, hydrogen is determined as H_2O, and nitrogen species are reduced over hot copper to N_2 for detection by thermal conductivity. For sulfur determinations, samples are run in open ceramic boats. For liquid samples, an inert support is placed under and over the liquid in the boat. For more refractory materials, vanadium pentoxide is added as a combustion accelerator. The sample is enclosed in tin capsules, burned in oxygen, and dropped into a tube packed with carbon black at 1140°C. For oxygen determination the carrier gas is nitrogen with 5% hydrogen. The oxygen present is quantitatively transformed to carbon monoxide, which is measured with a non-dispersive infrared (IR) photometer.

Samples

Form. Samples may be either liquids or solids.

Size. CHN analysis may be done either on the micro scale (3 to 10 mg per run), ideally in triplicate or the macro scale (25 to 250 mg per run), also in triplicate). Larger samples are always preferable for materials that may be inhomogeneous. Sulfur requires 25 to 200 mg per run, and analyses are performed at least in duplicate. Oxygen is always analyzed on the micro scale (3 to 10 mg per run) in triplicate.

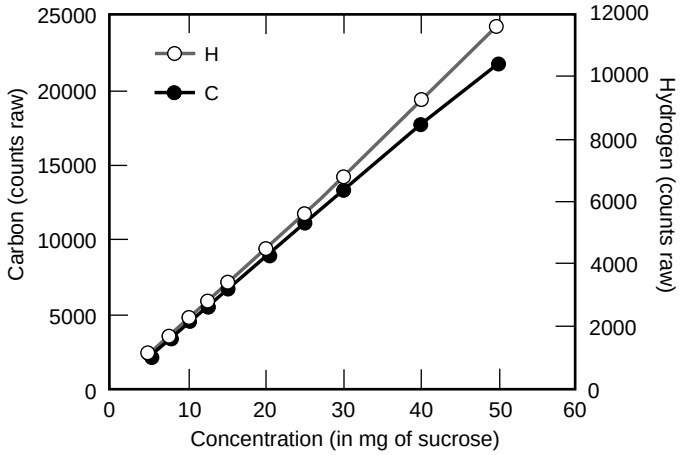
Preparation. Inorganic solids must be ground to fine powders before analysis. Special arrangements can be made for handling hygroscopic or air-sensitive materials.

Limitations

The method is destructive. In some cases, the presence of halogens will interfere in the analysis. It is important that the analyst be aware of their presence in planning the analysis. Highly refractory inorganic materials (e.g., metals, some minerals) may not be sufficiently combusted to give accurate results.

Examples of Applications

- Analysis of both organic materials and inorganic materials such as graphite, minerals, metal powders, and glasses.
- Analysis of raw and spent oil shale, oils, pure organic compounds, polymers, high explosives, oil shale process waters, aerogels, organometallic compounds, and coal.



CHN 800 cell linearization determination of total carbon in sucrose.

There are several handling problems. Strong acid solutions attack the tin or copper sample capsules. It may be impossible to seal very volatile organics into the capsules well enough for good results.

The limits of detection are about 0.3 wt% for all elements. Results can be obtained at lower values, but quantitation is poor.

Estimated Analysis Time

CHN Analysis

Macro analysis requires about 1 h to calibrate the instrument and then 5 to 10 min per run. Micro analysis requires 2 to 3 h for instrument calibration and 20 to 30 min per run. All samples are analyzed in triplicate.

Sulfur Analysis

Calibration requires about 1 h and each run takes 10 min.

Oxygen Analysis

This analysis requires that the instrument be reconfigured from the normal CHN mode and flushed with sweep gas for at least 24 h before calibration. Calibration is difficult, and accuracy and precision are not better than 5%, relative.

In all cases, analyzing multiple samples is more efficient because a single, daily calibration per day is usually sufficient. Samples with either very high or very low concentrations of the element to be determined may require special calibration and instrument conditions and will, accordingly, be more time consuming. Very small samples also require special handling.

Capabilities of Related Techniques

Low-level organic carbon can be determined (especially in aqueous solutions) by automated total organic carbon analysis.

Acid CO_2 determination may be combined with the total carbon analysis by CHN to yield values for organic and inorganic carbon in materials such as coal, shale, or minerals.

C, H, N, S, and O can also be determined in metals at parts-per-million levels by means of other automated equipment in the Plutonium Analytical Laboratory.

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